STANDARD OPERATING PROCEDURE (SOP)

	SOP NO.15	**EHS/SS-IM/SP /SOP-15**	**Effective Date**	**01-09-2022**	**REV. No.**	**00**
	SOP DESCRIPTION	**Chemical Analysis of Iron Ore fines,Base mix ,Mix material,Mill scale & LD Slag**	**SECTION**	SP Lab.		
	DEPARTMENT	Scientific Services - Iron Making	**PAGE**	**Page 1 of 13**		

Step No	Details of Activity (WHAT)	Associated safety Hazards/ Env. Impacts/ Quality Requirements	Responsibility (WHO)	Safe process (tools & Equipment, PPEs, Training etc.) (HOW)	Remarks / Reference (Document & Records)
01	**Chemical Analysis of Iron Ore fines,Base mix ,Mix material,Mill scale & LD Slag**	**SAFETY HAZARD:** **1-Burn injury**	**Shift In charge**	**PPE :** • **Heat prove Hand gloves** • **Safety goggles.** • **Safety Shoe.** • **Nose mask** • **Apron**	**IS 1493-2006** **IS 9959-1981**
		ENVIRONMENT: **1-Water pollution** **2-Air pollution**			**1.Chemical reagents at the time of normality preparation, chemical should be AR/LR make.** **2.Chemical should be kept in proper place.** **3. All the chemicals should have material safety data sheet(MSDS).**
		QUALITY: **1-Maintion Good housekeeping inside lad.** **2-Callibration required by NABL accredited Lab.**			

PREPARED BY	APPROVED BY	
Name and Designation :	Name and Designation :	
Signature with Date :	Signature with Date :	

Safe process:

1. Determination of Silica :

0.5 gm of dried sample shall be dissolved in 30 ml of Conc. HCl and is allowed to digest. It is then baked and cooled. 10 ml of 1:1 HCl and 50 ml of distilled water shall be added and boiled. Allow the residue to settle. The residue is filtered through whatman 41 filter paper/filter pulp wash with water till free from acid and the residue with filter paper taken in a platinum crucible (if fluorination is required) or in a silica crucible and is ignited at 950 deg C in muffle furnace which is then cooled and weighed (W1). The filtrate is collected in 250 ml standard flask and is used for further analysis (mother filtrate).

- **Fluorination**

Few drops of Conc. Sulphuric acid and 10 ml of Hydrofluoric acid shall be added to the ignited residue and is evaporated to dryness. The residue is then ignited in a muffle furnace for 5 minutes at a temperature of 950 +/- 15 deg C that is then cooled and weighed (W2).

% Silica = (W1-W2) X 100

where (W1-W2) = loss in weight in grams of the residue

The mass remaining in the crucible shall be fused with fusion mixture viz., sodium carbonate, borax and potassium carbonate, which is then leached with Hydrochloric acid and added to the mother filtrate obtained above in 250 ml standard flask.

2. Determination of Total Iron:

Reagents:

Dilute Hydrochloric acid (1:1)

Stannous Chloride solution 6% – Dissolve by heating 30 gm of stannous chloride in 30ml of distilled water and add 20 ml of conc. HCl

Cool and Preserve the solution in an airtight amber colored bottle to prevent oxidation.

Mercuric chloride solution – 6 %

Sulphuric – phosphoric acid mixture – add 375 ml of sulphuric acid to 1750 ml of distilled water and then add 375 ml of phosphoric acid and cool.

Barium diphenylamine sulphonate indicator – dissolve 0.32 gm of Barium diphenylamine sulphonate in 100 ml of hot water and add 10 ml of conc. Sulphuric acid.

Standard potassium dichromate solution (0.1N) – 0.1 N ampoule is made up to 500 ml mark in standard 500ml measuring flask or dissolve 4.9032 gm of potassium dichromate AR salt in one litre of distilled water. The solution shall be standardized with standard thiosulphate solution or freshly prepared ferrous ammonium sulphate solution.

Take 0.1 gm equivalent of solution from the mother filtrate add conc. HCl and boil. Add stannous chloride drop by drop until the color disappear, then add 10 ml of mercuric chloride. Wait till silky white precipitate appears then add sulphuric acid phosphoric acid mixture and 4 to 5 drops of Barium diphenylamine sulphonate indicator and dilute to 200 ml with distilled water. Titrate against 0.1N potassium dichromate and the end point is the appearance of violet color.

$$\% \text{ Total Iron} = \frac{\text{volume of dichromate consumed} \times 0.5585}{\text{No. of gm equivalence taken (0.1)}}$$

$$\% \text{ of Iron Oxide}(Fe_2O_3) = \text{percentage of Iron} \times 1.4289$$

3.Determination of Alumina (By EDTA method)

Reagents:

1:1 HCl

NaOH pellets

0.01 EDTA solution: 3.72 gm of EDTA salt dissolved in one litre of distilled water.

0.01 Zinc acetate: 2.19 gm of AR Zinc acetate dissolved in one litre of distilled water.

Sodium acetate buffer/ Ammonium acetate salt : 43 gm of sodium acetate dissolved in 500 ml of distilled water and 2 ml of glacial acetic acid is to be added.

Xylenol orange indicator

Take 50 ml from mother solution into 250 ml beaker and warm it. Add NaOH pellets till iron precipitated. Warm the solution to 2 to 5 minutes to settle the precipitated. Filter the solution in 500 ml conical flask by using No. 41 whatman filter paper. Add known volume excess EDTA solution (0.01M). Make the solution slightly acidify by adding 1:1 HCl drop wise. Add 10 to 15 ml of sodium acetate buffer or 10 – 15 gms of Ammonium Acetate salt to the solution for pH and just boil and cool. Titrate the solution with 0.01 M Zinc acetate by using Xylenol orange as indicator. End point is yellow to red.

$$\%\ Al_2O_3 = \frac{(\text{Volume of 0.01 M EDTA added} - \text{Volume of 0.01 M Zinc acetate consumed}) \times 0.05101}{\text{Weight of sample}}$$

4.Determination of Phosphorus:

Take 2gm of dried sample in a 500ml beaker add 50ml of CONC HCL for dissolving & keep it on hot plate for pasty condition. Then add 10ml of Conc. HNO3 & heat it till paste condition. After cooling redissolved with conc. HNO3 & boil for 5-10mints.Then filter in a conical flask with 41 filter paper. Take filtrate boil for 5-10mints & add 2-3 drops of KMnO4 solution & boil till the colour stays.Then add 2-3 drops of NaNo2 solution. Add Ammonia solution till iron precipitated, the same precipitate redissolved in Conc. HNO3. Add excess HNO3 to maintain the solution acidic. Then heat up to 70 deg C. Then add Ammonium Molybdate solution & shake vigorously till yellow ppt obtained. Keep the solution for 4 hrs for settled.

Reagents:

Dilute ammonium hydroxide – 1:1

Dilute Nitric acid – 1:1 & 1: 100

Concentrated Nitric acid

Potassium Nitrate

Ammonium Molybdate solutions.

Solution A:

Dissolve 100 gms of Molybdic acid or 118 gms of Ammonium hepta molybdate in a mixture of 145 ml of concentrated ammonium hydroxide and 270 ml of water. Boil for a while and cool thoroughly.

Solution B:

Add 490 ml of concentrated Nitric acid to 1150 ml of water or 300 ml to 700 ml of water. Solution A is added to solution B with constant *stirring and filtered through number 1 filter paper before use.*

Potassium Nitrate wash solution – 1 %
Phenolphthalein indicator – dissolve 0.2 gm of phenolphthalein in 100 ml of 60 % alcohol.
Standard Nitric acid – 0.1 N Ampoule is made upto 500 ml mark in standard 500 ml measuring flask or take 7 ml of concentrated HNO_3made up to 1 litre with distilled water.

Standard sodium hydroxide solution: - 0.1 N Ampoule is made upto 500 ml mark in standard 500 ml measuring flask or 4.5 gm of NaOH dissolved in 1 litre distilled water.

The precipitate is filtered with whatman 40 filter paper pulp and the filter paper pulp is washed with potassium nitrate solution. The washing is given till 10 ml of the filtrate collected in a test tube does not consume more than one drop of 0.1N sodium hydroxide solution using a drop of phenolphthalein indicator solution. Transfer the precipitate with pulp to the conical flask and add distilled water. 10 ml of standard sodium hydroxide solution and 3-4 drops of phenolphthalein indicator is added to the conical flask. Titrate with standard nitric acid until pink color is just discharged. A blank is carried out with the same quantity of sodium hydroxide solution, which was added in the test and titrated with standard Nitric acid.

$$\% \text{ of Phosphorus} = \frac{(A-B) \times 0.0135 \times 100 \times C}{D}$$

Where

A - Volume of HNO_3 required for Blank

B - Volume of HNO_3 required to titrate excess sodium hydroxide.

C - Normality of HNO_3

D - Weight in gram of sample in aliquot taken.

5.Determination of Loss on Ignation:

1 gm of test sample shall be weighed accurately in a platinum / Silica crucible. The crucible shall be heated gently at first and finally at 950 to 1000 deg C for one hour. It is then cooled and weighed until constant weight is obtained the process of heating continued.

$$\% \text{ of loss on ignition} = \frac{(W1 - W2)}{W3} \times 100$$

Where

W1 - weight of sample and crucible before ignition

W2 - weight of sample and crucible after ignition

W3 - weight of sample taken

6. Determination of FeO:

0.5 gram of sample shall be taken in a conical flask with sodium bicarbonate and moisten with some drops of water and 20ml of hydrochloric acid is added and an inert atmosphere is maintained by sealing the conical flask with a cork. Contents with conical flask are boiled and cooled. 30 ml of acid mixture (phosphoric acid and sulphuric acid) and two drops of Barium diphenyl amine sulphonate indicator shall be added and titrated with 0.1N Potassium Dichromate solution and the end point is indicated by appearance of violet color.

$$\% \text{ of Ferrous Oxide (FeO)} = \frac{A \times 0.5585 \times 1.286}{B}$$

Where

A - Volume of 0.1 N Potassium Dichromate consumed

B - weight of sample taken

7.Determination of Calcium Oxide: (EDTA method)

Reagents:

Glycerol.

Triethanolamine.

4N sodium hydroxide: Prepared by dissolving 160 gms of sodium hydroxide pellets in 1 litre of distilled water.

Patton & Reeder's Indicator

0.1 M EDTA solution: 3.72 gm of EDTA dissolved in 1 litre of distilled water.

10 ml of Aliquot shall be taken from the main filtrate in a 400 ml beaker having 100 ml of distilled water. 5 ml of glycerol and 2 ml of triethanolamine and 4N sodium hydroxide shall be added to this to adjust the pH at 12 or slightly more. A pinch of Patton readers indicator shall also be added to this for titration against 0.01M EDTA until the color of the solution changes from wine red to greenish blue.

% of CaO = A X 0.2802

Where A = volume of 0.01 M EDTA consumed.

8. Determination of Magnesium Oxide (EDTA method):

Reagents:

Triethanol amine

PH 10 buffer: prepared by adding 560 ml of liquid ammonium hydroxide and 68 gms of Ammonium chloride. To this, 440 ml, distilled water shall be added.

Eriochrome – black T indicator

0.01 M EDTA solution

10 ml of aliquot shall be taken from the main solution into a 400 ml beaker. About 5 ml of Triethanolamine 20 ml of buffer solution shall be added with constant shaking. A pinch of Eriochrome – black T indicator shall be added and titrated against 0.01 M EDTA solution until color changes from wine red to blue.

% of Magnesium Oxide = (B – A) X 0.2016

Where,
B - Volume of EDTA consumed MgO and CaO titration
A - Volume of EDTA consumed CaO titration

9. Determination of Na2O & K2O:

Preparation of Standard Solutions for Calibration of the instrument:-

Prepare 1000ppm of 'Na' & 'K' Solution by dissolving 2.54gm of dried NaCl & 1.9067gm of dried KCl.
Take required amount of 1000ppm solution to make 100ppm solution.
From 100ppm solution, prepare 5, 10, 15,20,30,40 ppm solution.
1ml of 100ppm in100ml of distilled water =1ppm solution.

Calibration of the Instrument & testing sample:-

1. Switch on the instrument.
2. Switch on the compressor, set at 0.5kg/cm sqr. Run it for 5 minutes to clear the automiser chamber with distilled water.
3. Open the knob of gas regulator of the cylinder and light up the burner and set it at 81-83mv.
4. Calibrate the instrument by running standard solutions of 5, 10, 15, or 20, 30, 40 as per our requirement.
5. Run the Sinter standard and note the reading to know the exact factor.
6. Run the samples and note down the reading.
7. Sinter standard sample prepared by dissolving 0.5 grms in HCl, baked and SiO2 removed and made up to 250ml.
8. Test sample is to be prepared as sinter standard sample preparation.

Calculation:-

%Na2O=Reading-Blank x 1.348 x factor x 100/ sample wt. X 1000000

%K2O=Reading-Blank x 1.2046 x factor x 100/ sample wt. X 1000000